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FIREFIGHTING AND EMERGENCY RESPONSE STUDY OF ADVANCED COMPOSITES AIRCRAFT

Objective 4: Post Fire Decontamination of Personal Protection Equipment

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14. ABSTRACT
Concerns existed regarding the decontamination of personal protective equipment (PPE) exposed to fires involving advanced composite materials (ACM). The concerns focused on soot particles and fugitive fiber emissions produced from burning ACM and expected to deposit on turnout suits and equipment. Contaminants to PPE were studied from live ACM fires and from contact with burned ACM. Photomicrography techniques were developed to provide quantitative measures of the contaminants. MATLAB and the Image Analysis Toolbox were used to quantify particulates photographed through microscopes on PPE material and test coupons. Coupons of material used in proximity bunker suit construction were exposed to burned ACM and were then cleaned, with quantitative measures of surface particle contaminants made prior to exposure, after exposure, and after cleaning, to test cleaning techniques of water washing, vacuum cleaning, simple brushing, decontamination with wipes, and cleaning with a sticky lint roller. Water washing and a sticky lint roller were the best of the techniques examined for removal of ACM-derived particulate contamination.

15. SUBJECT TERMS

composites, advanced composite material (ACM), microscopy, MATLAB, cleaning, decontamination

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FOREWORD

In 2009 the Air Force Civil Engineering Support Agency (AFCESA) commissioned the Fire Research Team of the Air Force Research Laboratory, Airbase Technologies Division (AFRL/RXQ) to perform a four-pronged research and development effort to better understand and fight fires involving composites and composite aircraft. Four objectives were developed: (1) Damage Mitigation from Small Fires, (2) Firefighting Effectiveness of Technologies and Agents on Composite Aircraft Fires, (3) Penetrating and Overhauling Wreckage, and (4) Post Fire Decontamination of PPE and Equipment. This report documents experiments and findings from Objective 4 – Post Fire Decontamination of PPE and Equipment.

PREFACE

Support for this investigation was provided to AFRL/RXQ from AFCESA. The interest by AFCESA and AFRL stemmed from difficulties encountered in recent aircraft accidents involving aircraft which incorporated large amounts of composite material in their structure, as well as historic concerns. The authors were assisted by additional members of the AFRL/RXQ Fire Research Team. Dr. Doug Dierdorf suggested the "bake and shake" technique to contaminate fabric and equipment samples with particulates from post-fire burned composites. Professional firefighters William Fischer and John Patnode conducted controlled research fires to assist the authors. Ms. Jennifer Schroeder arranged for a generous supply of fabrics used in preparing bunker uniforms. 1Lt Eileen Shannon and Ms. Karen Farrington provided expertise and advice regarding photomicroscopy, a technique that proved critical in this investigation. McCrone Associates and Dr. Benham Poudremi of the University of North Carolina also examined selected materials and offered advice.

1. SUMMARY

Experiments were conducted to evaluate decontamination and cleaning techniques for firefighting equipment exposed to smoke and fugitive fiber emissions from composite material fires. Particulate emissions from composite fires offer dermal and respiratory threats and contaminate firefighting equipment including personal protective equipment (PPE). Cleaning experiments were performed and determined water washing or cleaning with sticky lint rollers cleaned PPE to levels not significantly different than the original items. Further these techniques cleaned better than brushing or cleaning with decontamination wipes. Vacuum cleaning was shown to be slightly less effective than water washing or cleaning with a lint roller, although it could be recommended in some situations.

2. INTRODUCTION

2.1. Composite Fire Hazards

Fires involving composite aerospace materials have been suspected as a source of unusual hazard. The hazards were originally suspected as a source of contamination and a danger to electronic equipment from fugitive reinforcing fibers released as the polymer matrix of the composite burned. In response, the National Aeronautics and Space Administration (NASA) initiated a study of reinforcing fiber release due to combinations of burning and impact from graphite-epoxy composite, graphite-Kevlar, glass-graphite, and boron-graphite hybrid composites. The results were inconclusive regarding threats to electronic equipment.[1] Follow-on experiments were performed to expose operating electronic amplifiers to cut fibers and fibers from burning composite, to gauge the dangers composite smoke and fugitive fibers presented toward electronics and avionics. Vulnerabilities to fibers were measured through failure of exposed stereo amplifiers, which were used as surrogates for more valuable electronics. The exposed amplifiers survived exposure to soot and to soot with glass fibers but failed with measurable time-to-failure when exposed soot and carbon fibers from shock tube experiments. The failure rates of the stereo amplifiers from burning graphite-epoxy composite were similar to those expected for amplifiers exposed to cut virgin fiber.[2]

Composite aircraft accidents have generated reports of skin and respiratory irritation among firefighters and post-crash recovery personnel. A Royal Air Force (RAF) Harrier GR5, containing carbon/epoxy composite crashed in Denmark in 1991. The recovery team suffered eye and skin irritation and respiratory difficulties.[3] Firefighters responding to the 1997 crash of an F-117 reported nausea, headache, eye and skin irritation, and respiratory difficulties on exposure to the smoke from the aircraft.[4] The emissions from burning composite are believed to generate dangerous materials in the form of toxic vapors and gases, smoke particles and fugitive fibers.[5] A team (Courson *et al.*) from the Armstrong Laboratory Toxicology Division collected and analyzed smoke from burned composite. They measured smoke particle diameters and extracted, identified, and quantified a number of organic compounds, some of which were toxic or carcinogenic and some suspected of such threats. Their study did not identify or examine fugitive fibers.[6]

A large-scale observation of burned composites was conducted at the AFRL's Tyndall AFB operating location. This involved a complex team from the Air Force Institute for Environmental Safety and Occupational Health Risk Analysis, AFRL, the Mississippi Air National Guard, and the RAF to burn sections of composite wings, collect samples from the combustion solids and gases, and sample occupational threats encountered by post-fire recovery personnel. Both NIOSH-style occupational health sampling and EPA-style particulate collection equipment were adapted to sample smoke particulates in this study, and special emphasis was placed on particles in the 1-2.5μm diameter range. Vapor-phase organic compounds were collected with solid-phase microextraction samplers (SPME) and qualitatively evaluated by gas chromatography/mass spectrometry (GC/MS).[7] Much of the collection equipment used for the combustion solids was designed with the concept that the particles would be roughly spherical in shape. Therefore, it is not known whether the sampling systems used in this study exhibited a bias against long particles.

Advanced composite material (ACM) fires produce aerosol smoke, vapor-phase and gaseous chemicals, and fugitive fiber fragments. Fugitive fiber fragments are believed to originate from the reinforcing fibers in the composite, freed from the bulk material as the resin burns away. The fugitive fibers are the unique threat from composite material fires, as smoke and gases are present in any fire.[5] Occupational and environmental safety concerns focus considerable attention on fugitive fibers. Hertzberg conducted small sample captive burns to measure normal soot and fiber from graphite-epoxy composite fires. Spherical soot particles dominated in number over fibers in his particulate catches. He investigated standard smoke production methods for small samples, including ISO standard 5659.[8] Graphite fibers are the most commonly discussed fibers, correlating with the common use of graphite-epoxy composites in aerospace materials.

Ghandi and Lyon identify two primary exposure routes from fibers – dermal exposure and inhalation. Dermal exposure was deemed most dangerous to the unprotected eye, though the dermal irritation was not expected to be permanent. Inhaled fibers could cause irritation and even more serious effects throughout the respiratory tract. Few toxicology investigations have focused specifically on reinforcing fibers such as graphite or polyacrylonitrile (PAN). Considerable toxicology efforts have focused on asbestos fibers, and some discussions have substituted asbestos data for incomplete data from ACM fibers.[5]

Additional inhalation studies with non-ACM fibers have been conducted. Okabe *et al.* exposed hamsters to nasal inhalation of fiberglass fibers and measured the aerosol concentration and breath parameters. The subject animals were sacrificed and the lungs and trachea were removed, digested, and analyzed to measure and quantify the fibers deposited in the respiratory tissues. The authors found approximately 25% of the inhaled fibers were deposited in the hamster lungs and trachea, with some variation with respiration rate – the fiber deposition appeared to vary inversely with respiration rate, but to a weak extent.[9] Raymann-Rasmussen *et al.* investigated the effects of carbon nanotubes inhaled by mice. The carbon nanotubes were shown to be able to reach the lung tissues and cause lung irritation effects. More serious effects such as cancer were not demonstrated.[10]

Whitehead *et al*. investigated acute lung injury (ALI) physical and biochemical parameters in rats exposed to smoke from graphite epoxy ACM combustion and compared with the parameters exhibited by control rats injected with paraquat dichloride. ALI was documented from ACM smoke exposure and the paraquat positive control by monitoring several biochemical markers for inflammation over the course of up to seven days post-exposure. Several subject animals also died in the immediate post-exposure period, from both the smoke exposure and the paraquat injections although the precise cause was not determined.[11]

Human experiments cannot be made using live subjects and sacrificial techniques, but several studies investigated deposition of fibers in models of the human respiratory tract. Zhou *et al*. prepared a replica of a human airway and examined deposition of carbon fibers which varied in length but possessed nominally constant diameter, reporting on the deposition in the various regions as a function of Stokes number, used as a measurement for the effective particle size.[12] Su and Cheng performed similar studies exclusively on a replica of the human upper nasal airway.[13] Wang *et al*. utilized a similar model and varied flow rate parameters.[14]

2.2. Contamination in an Operational Event

On 23 February 2008 a B-2 aircraft crashed on take-off from Andersen AFB, Guam. The airframe utilized large quantities of ACM and was largely consumed in the resulting fire. The Andersen fire department experienced difficulties in extinguishing the fire, having to call in off-duty firefighters and firefighters from neighboring facilities. The fire required a larger-than-anticipated quantity of water and firefighting agent to extinguish and raised issues regarding appropriate firefighting techniques and agents for fires involving ACM.[15] Reports from Andersen AFB and the AFRL Advanced Composites Office indicate the fire department and aircraft maintenance squadron suspected contamination on their PPE clothing and equipment. Lacking clear guidance on decontamination of the respirators and protective clothing used in ACM fires, all such equipment was reportedly discarded and replaced. The experiments reported herein intend to investigate cleaning techniques for use in future ACM fires. These experiments were intended to satisfy questions posed to the AFRL/RXQ Fire Research Team by AFCESA, i.e. to determine satisfactory decontamination procedures for PPE and other equipment.

2.3. Planned Experimental Strategy

Fabric from firefighters' PPE and related equipment were exposed to composite material fires and secondary contaminants such as contact transferred soot and fugitive fibers. The contaminated gear was then cleaned and an evaluation was made to measure which cleaning techniques cleaned the equipment best, and if any of the studied techniques could clean the equipment to initial "clean" conditions.

The basic firefighter PPE exposed to contamination was the bunker gear or turnout suit. Bunker suits in the experience of the authors come in two types, close approach bunkers (also termed proximity suits or silver suits) used when the firefighters must work in very close or direct contact with burning materials, and structural bunkers used for more common fires in buildings (or structures). Both types of bunker suits are highly insulated but proximity bunkers add an outer layer of fabric covered in a final silvered reflective layer to reflect heat away from the wearer. Proximity suits are commonly worn by firefighters responding to aircraft fires, where the firefighters may have to approach the burning aircraft to rescue passengers and crew or to cut access ports into the aircraft to fight concealed fires. Proximity suits are also encountered as PPE in metal factories and in sampling from recently erupted volcanic material (lava sampling). Proximity bunkers have been the most commonly used turnout suits for fighting aircraft fires and they are expected to remain so.

3. METHODS, ASSUMPTIONS, AND PROCEDURES

3.1. Live Fire Sampling

Composite burns were conducted to support two objectives requested by AFCESA, Objective 2 and Objective 4. Objective 2 was reported separately (AFRL-RX-TY-TR-2011-0047) and contains additional details on live fire burns.[16] Live fires were conducted in the AFRL/RXQ Fire Hanger facility on Test Range II, Tyndall AFB, FL. The live fire experiments were designed to duplicate aircraft accident burns by kindling a jet fuel (JP-8) pool fire under and adjacent to a composite sample that was placed under mechanical stress by elements of the sample support. These fires were conducted in the Fire Hanger (Figure 1), a closed but non-airtight shelter. Samples of firefighter uniform material were exposed to the smoke and gases from these fires by placing arrays of material coupons near enough to the fires to experience the smoke but far enough away to avoid thermal damage.

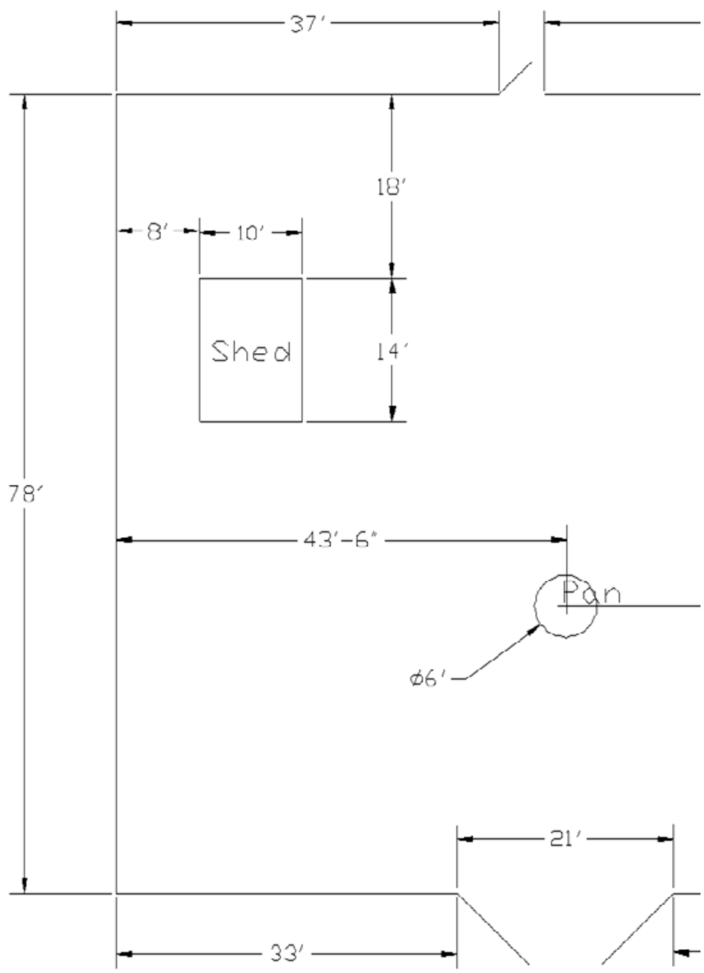

Figure 1. Floor Plan of the Fire Hanger, Showing the Location of Doors, the Fire Pan, and a Metal Instrumentation Shed

3.2. "Bake and Shake" Technique

An alternative method to "soot-up" samples was to expose coupons of experimental material to soot, fibers, and other particulates from previously burned composites. A supply of burned composite material was collected after a fire on 23 Nov 2009. A 5-gal bucket with lid was filled with burned composite material which had been wetted but had no fixant applied. An exposure chamber was improvised from a Bankers Box® (StorFile™ 799) with an added corrugated cardboard sheet cut to fit flat on the bottom of the box and trimmed to fit. The coupons of suit material and burned composite were attached to the cardboard sheet in a rectangular array using sewing pins inserted through the center of each coupon. The coupon sheets were then placed in the exposure chamber and agitated to contaminate the coupons.

The original composite was carbon/epoxy, with 1-inch tape in a 0/90 layup. As this burned, strips of material corresponding to the original tape width separated from the composite bulk and fell off. The burned composite material consisted primarily of strips of material, approximately 1-inch wide and arbitrarily long, with several layers of reinforcing fibers alternating with binding resin. For these experiments, longer strips were cut into shorter segments, 1-2 inches long, to facilitate weighing and better distribute the composite material and associated contaminants throughout the exposure chamber. Composite strips used to expose experimental coupons were weighed in a beaker, using an analytical balance (Mettler AE-200) prior to use in the exposure chamber.

Adjustable parameters and details of the cleaning experiments are given in Section 4.3.

3.3. Photomicrography for Detection of Contaminants

Many of the soot particles and fibers were visible with the unaided eye, but when viewed through a microscope they could be seen better and size and number measurements were permitted. Camera-microscope instruments were utilized to photograph the contaminants and support quantification of the contaminants by analysis of the images.

A handheld digital microscope (Celestron™ model 44302) was used for moderate power imaging of samples with overhead LED lighting. The microscope connected with a computer via a USB 2.0 interface for communications and power supply. The microscope offered two magnification ranges, 10X-40X (dependent on distance from the sample), and 150X. Overhead illumination was provided by an array of LEDs arranged around the objective lens. The microscope was controlled through Digital Microscope Suite 2.0 software (Celestron) operated on a laptop computer (Dell Latitude 610). The laptop displayed images from the digital microscope and thus substituted for the eyepiece of a conventional microscope. Images selected for storage were captured on command from a "shutter button" on top of the microscope or by selection of a command button on the computer display window. Captured images were stored as 1280 X 1024 pixel (1.3 MPixel) digital image files in Joint Photographic Expert Group (.jpg) format. A metal stand was provided to assist in positioning and stabilizing the microscope.

The handheld microscope was useful for direct imaging of intact PPE and coupons for deposited solid contaminants. Greater control of focus and magnification was exhibited for coupons, but the handheld microscope could be used to image particulates on equipment even while being worn. Direct imaging was used to examine contaminants on firehose samples returned from the 2008 B-2 crash at Andersen AFB. Direct imaging was also used to examine soot and fibers on coupons of bunker fabric. Two bunker fabric materials were of initial interest, silver colored material from proximity bunkers and the brown-orange fabric material used as the outer layer of structural bunkers.

Slide-mounted samples could be examined with a triocular microscope (Southern Precision Instruments, SPI 1864) equipped with dual 10X eyepieces and an auxiliary optical tube for camera mounting. An auxiliary light source (Dolan-Jenner Industries, Inc, Fiber-Lite Series 180) provided high intensity overhead light from either side of the microscope objective lens and permitted imaging of samples laying on the slides, on the stage, or affixed to the bottom of the slide. The digital camera (Motic, Moticam 2500) mounted on the auxiliary optical tube and was interfaced to the laboratory laptop computer (Dell 610 also used for the Celstron microscope) via a USB 2.0 interface for control of the camera and storage of the images. Motic Images 2.0 was the software used to acquire, store, and organize the photomicrographs. This software stores images in a proprietary format with an .sfc file extension, but could translate the images into a .jpg format. The .sfc images were stored with the maximum resolution available from the Motic software, 2592 × 1944 pixels, but .jpg translations were only stored with 1280 × 1024 pixel resolution.

Some fabric coupons were examined with the SPI microscope and Motic camera, but the narrow plane of focus for this combination caused the system to yield poor images unless the samples were constrained to a flat viewing field, a condition usually best satisfied by mounting the sample on a microscope slide. A set of exposed coupons were submitted to McCrone Associates for evaluation, and they recommended sampling the particulates from the fabric samples with sticky pad media and transferring the sticky sheets to microscope slides. Acting on this suggestion, 18-inch × 36-inch 30-sheet sticky pads were obtained for sampling. One set of pads were “clear-on-white” and were clear sheets on a white final backing sheet (Miller Products Co, XTRACLEAN™, product no. CSS1836-30F-C/W). Opaque white sheets were also obtained as 30-sheet tear off mats of 18-inch × 36-inch sticky sheets (American CleanStat, Tackymat, product no. 183602WW-460). Both types of pads could be cut into 1-inch squares, to match the dimensions of the microscope slides.

To obtain a sample, the top, non-sticky, sheet was removed from the 30-sheet pad to expose a sticky sheet. This sheet was pressed on to a sample coupon to adhere particulates and then placed onto a clean microscope slide (Diagger frosted, precleaned microslides, catalog no. EF15975F). Each slide was labeled as to the sample contents by writing on the frosted end. The remaining sticky pad was freed by pulling off the top sheet which held the sampled particulates trapped between the sticky side of the sheet and the glass slide. Pulling off the sampled sheet exposed the next layer of the sticky pad to collect the next sample. The mounted slides could then be examined with the microscope-camera combination. The best quantitative results obtained for this study were from particulates sampled (trapped) with opaque sticky sheets, affixed to the bottom of a slide, and viewed through the slide. This forced the entire sample into a restricted focal plane, preferred for focusing the microscope, and protected the sample from alteration by external contamination or inadvertent removal of the particulate catch.

The photomicrographs were analyzed under the assumption that dark areas or objects on a generally light background would indicate particulate contamination. MATLAB version 2008b or later versions and the Image Processing Toolbox (Mathworks, Inc., Natick, MA) were used to analyze the images. MATLAB did not support analysis of .sfc files but could open and analyze .jpg files. The MATLAB Image Processing Toolbox provided routines to open .jpg image files, display images, convert color images to grayscale images (analogous to scenes on a black-and-white television), and detect objects in binary images including counting the objects and measuring the total area (the number of pixels) of all objects in an image. The photomicrograph images were converted from color images to grayscale images using the relationship in Equation 1. Grayscale images were converted to binary black (value 0) and white (value 1) images through the use of a user-supplied threshold value. An interactive application was developed to display the original image along with a binary converted image, and allow the user to enter a threshold value through a graphical slider input. When the converted binary image matched the user's perception of the particulates in the image, a control button caused the application to calculate the number of objects in the binary image and the total area of all objects in the image.

$$\boldsymbol{GS_{x,y} = 0.2989R_{x,y} + 0.5870G_{x,y} + 0.1140B_{x,y}} \tag{1}$$

Where $GS_{x,y}$ represents the grayscale value for the pixel at image coordinates x and y, $R_{x,y}$ represents the red value for the pixel, $G_{x,y}$ represents the green value of the pixel, and $B_{x,y}$ represents the blue value.

The interactive screen used to facilitate selection of the appropriate threshold is shown in Figure 2. When the MATLAB application was loaded the interactive figure was displayed with blank panes where the images would be placed. The "New Image" button was selected to load a .jpg file containing the original image to analyze. This image was then displayed in the upper image pane, and the application would generate a grayscale image from the original color image, as discussed above (Eq. 1).Next a loop process was started to convert the grayscale image to a binary image based on threshold values input by the user. The binary image was displayed after each conversion in the lower image pane.

The threshold variable in the application was set by a slider input, ranging in value from 0-255. The user adjusted the slider to change the threshold value which updated the binary conversion image and displayed it in the lower image pane. In some cases a compromise was necessary seeking a threshold high enough to allow the system to recognize some particles but low enough to prevent shaded areas, such as the corners of the image, from being misrecognized as particles. When the user was satisfied that the best conversion had been achieved the "Done" button was selected to calculate and display the results (threshold, particle count, and particle area) on the MATLAB command window. The application stored the results in an Excel-formatted spreadsheet and the user also copied the results manually to a spreadsheet to organize the data from the cleaning or smoke exposure experiments.

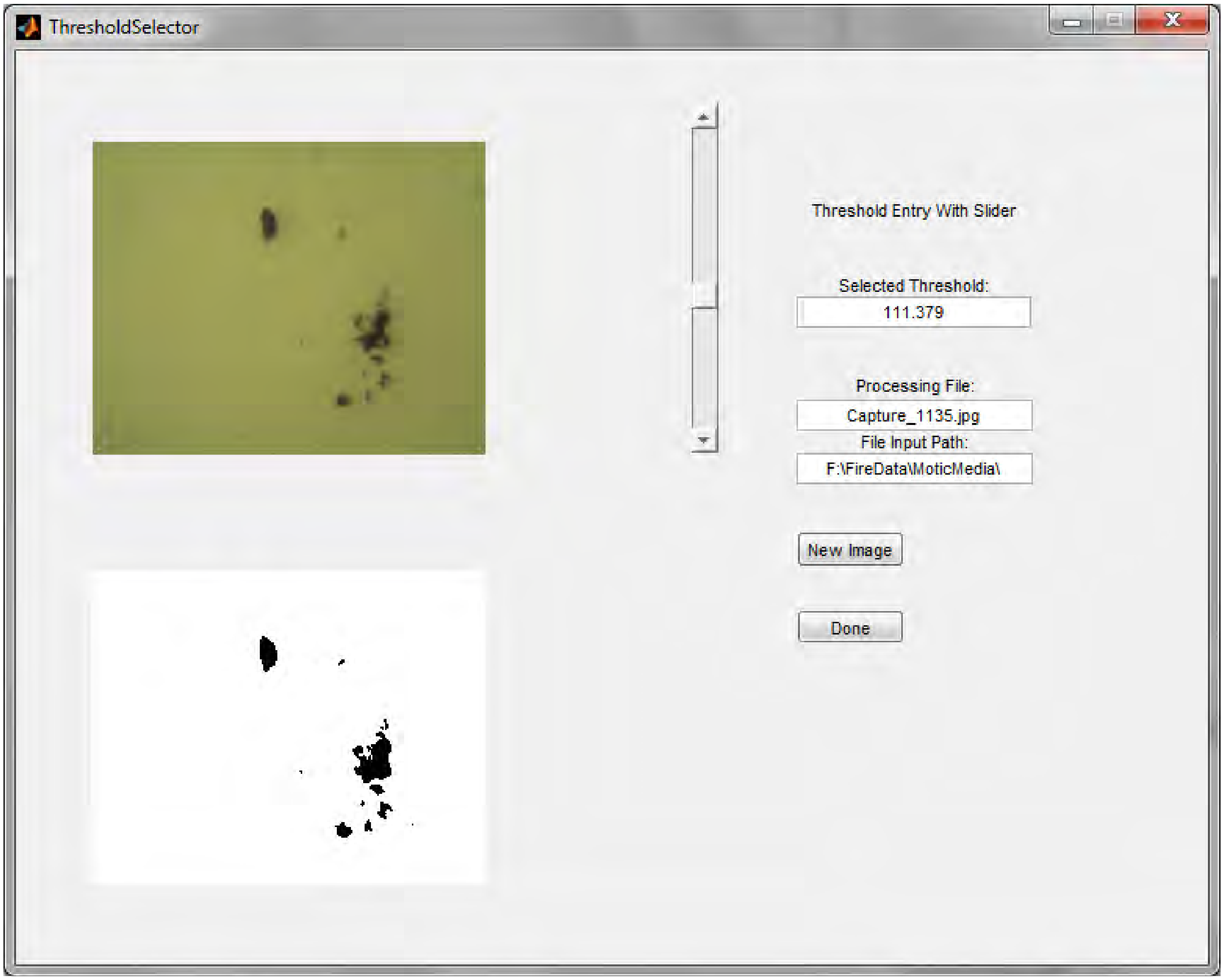

Figure 2. MATLAB Screen Used to Facilitate Selection of the Image Binary Conversion Threshold

*Note this binary image pane suffered some degradation from the actual screen when captured for this report.

3.4. Cleaning Methods

Coupons were cleaned by brushing, water washing, vacuum cleaning, wiping with decontamination wipes, or with a lint roller. Brush cleaning was accomplished with the brush-end of a vacuum cleaner wand with the vacuum cleaner shut off. In order to simulate washdown with a low-pressure fire hose, type II wash water was provided through a pressurized hose delivering water from a water purification system (Siemans, Inc.). Vacuum cleaning was accomplished with a HEPA-filtered shop vacuum (Ridgid® model E43484 wet-dry vacuum cleaner with VF5000 filter). Lysol® decontamination wipes (Lysol® 4-mil disinfecting wipes) were used for wipe cleaning, with each coupon being wiped on either side of the pin holding the coupon onto the sample array, then the coupon was rotated 90° and the coupon was wiped on either side of the pin again. Each area of the coupon should have been contacted with the wipe twice in this procedure. Coupons cleaned with the lint roller (Evercare 60 sheet UPN 70982-01072) were removed from the sample array and placed with the exposed, silver side of the coupon facing the sticky side of a sheet of the lint roller, then the coupon was removed and placed on a clean area of the sheet two more times, for a total of three exposures to the sticky cleaning sheet. Cleaning sheets were torn off after each coupon to expose a clean surface on the lint roller.

3.5. Statistical Treatment of Particle Measurements

Quantitative measurements were made from sticky pad samples affixed to microscope slides. Attempts were made to image nine fields of view (FOV) from each microscope slide. Precise locations of the FOV were not made and there was some variation in size and shape of the samples, but attempts were made to image a FOV in each of nine areas of each slide, eight along the edges (but far enough away from the edge so that the edges and associated handling contamination didn't contribute to the object counts) and an FOV near the center of each sample.

Each cleaning class was the set of samples prepared with a given cleaning technique. The MATLAB-based image analysis routine produced a count of the number of particles and area of the particles in each FOV. The areas of the particles were treated as descriptive statistics. Averages were calculated from the particle area over all fields of view for a cleaning class. The median was calculated as the median number of particles from all fields of view for a cleaning class. The number n given for each class was the total number of all fields of view for the cleaning class. The number of degrees of freedom, df, used for Students-t and other comparison testing, was calculated as the number of observations, n, minus a degree of freedom for each coupon.

Cleanability is a measure of the ability to clean. The concept was obtained from standards for cleaning aircraft paint topcoats. The concept was originally applied to light reflecting from a painted surface, where paint soiled with carbon black particles was expected to reflect less light.[17] The concept was used in this study because of the similar nature of this study. For this report the cleanability C was calculated from the measured area of smoke particles, using the formula in Equation 2.

$$C = \frac{(A_s - A_c)}{(A_s - A_o)} * 100\% \qquad (2)$$

> Where C represents the cleanability value for the material and cleaning technique, A_s represents the average particle area of soiled coupons for a sample class, A_c represents the average area of particles on the cleaned coupons, and $A_o$represents the average area of particles on original coupons, prior to the exposure of the burned composite.

The pooled standard deviations for each cleaning class were calculated as in Equation 3.[18] Each coupon, with 7-9 fields of view, were considered a sample set with associated number of degrees of freedom, v, equal to the number of fields of view less one and sample estimate of standard deviation, s.

$$s^2_{pooled} = \frac{\sum_{i=1}^{n} v_i s_1^2}{\sum_{i=1}^{n} v_i} \qquad (3)$$

Students-t tests were performed on the cleaning class means for significant difference between two sample means.[19] The experimental t-statistic was calculated as in Equation 4 and then compared with tabulated critical t-values for 95% confidence ($\alpha = 0.05$) and 90% confidence ($\alpha = 0.10$).

$$\boldsymbol{t} = \frac{(\bar{x}_1 - \bar{x}_2)}{s_{pooled}} \qquad (4)$$

The median number is the number in the middle of an ordered set or numbers – if the set is odd, the median is the central number and if the set is even the median is the average of the central two numbers.

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4. RESULTS AND DISCUSSION

4.1. Direct Imaging Microscopy

Direct imaging of the samples with the handheld Celestron™ microscope proved useful for examining bulk materials and larger samples that couldn't be placed on a microscope slide or easily positioned beneath the objective lens of the larger compound microscope. At the time samples were being examined with this technique, the variable magnification feature of the Celestron™ model 44302 left ambiguities regarding the actual magnification of some of the images. This technique and device did permit easy qualitative examination of samples.

The smallest object detectable to the image analysis system was an object of the size of a single pixel. The smallest object detectable at low power varied as the low power magnification varied with distance between the microscope and the sample. At the high power setting, the Celestron™ 44302 produced a pixel size of approximately 2µm.

4.1.1. Fire Hose Samples from Andersen AFB Crash and Fire

A set of cut fire hose sections was delivered to the AFRL/RXQ Fire Research Team from the February 2008 B-2 crash at Andersen AFB, and these were examined at low and high magnification with the Celestron™ microscope. The sections of hose varied in color and in degree of soiling. Direct imaging revealed a variety of particles trapped in the weave of the hose (Figure 3 and Figure 4). Some apparent soot particles were revealed for examination by the expedient of shaking out a section of hose on top of a clean kimwipe™ paper towel and then examining the particulates that dropped out with the handheld microscope (Figure 5 and Figure 6). The particles examined in this manner were revealed to largely be approximately spherical or rounded chunks of material. Essentially none of the longer rods or fibers were seen. Those were expected from release of the reinforcing fiber during burning of the composite resin. These hoses may have been used for incidents prior to the B-2 crash and fire, and contaminating material could be from the history of incidents and training events where these could have been used. Further some of the material may have been due to contact of the fire hoses with bare ground, i.e. ordinary dirt.

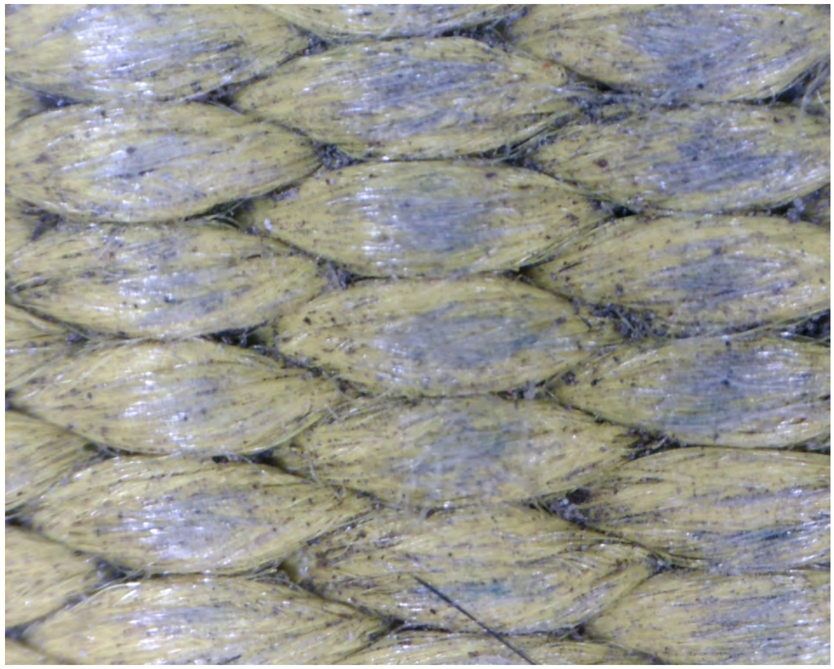

Figure 3. Low Magnification Image of Yellow Fire Hose

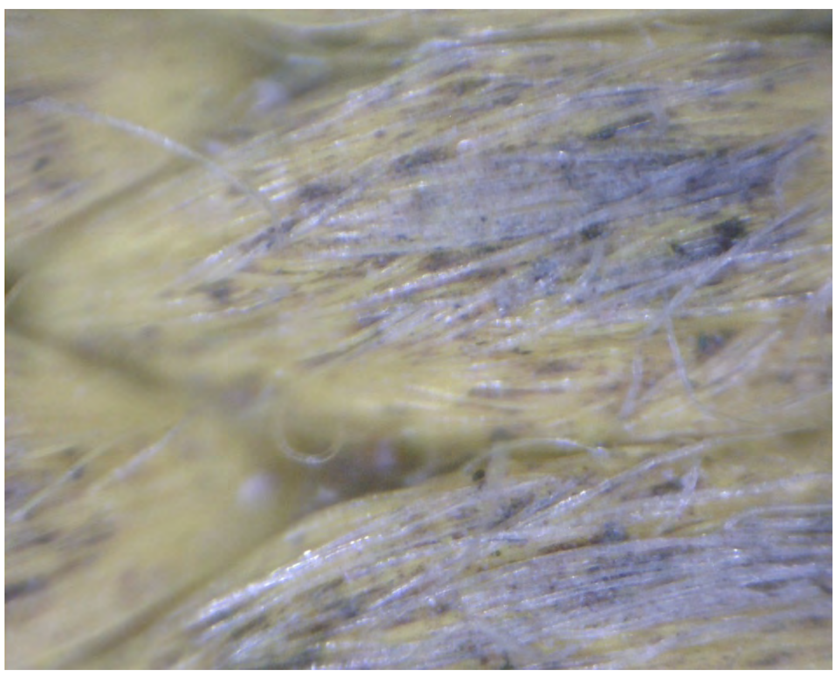

Figure 4. High Magnification (150X) Image of Yellow Fire Hose from the Andersen AFB B-2 Crash in 2008

Figure 5. Low Magnification Image of Particles Shaken from Andersen AFB Fire Hose Samples

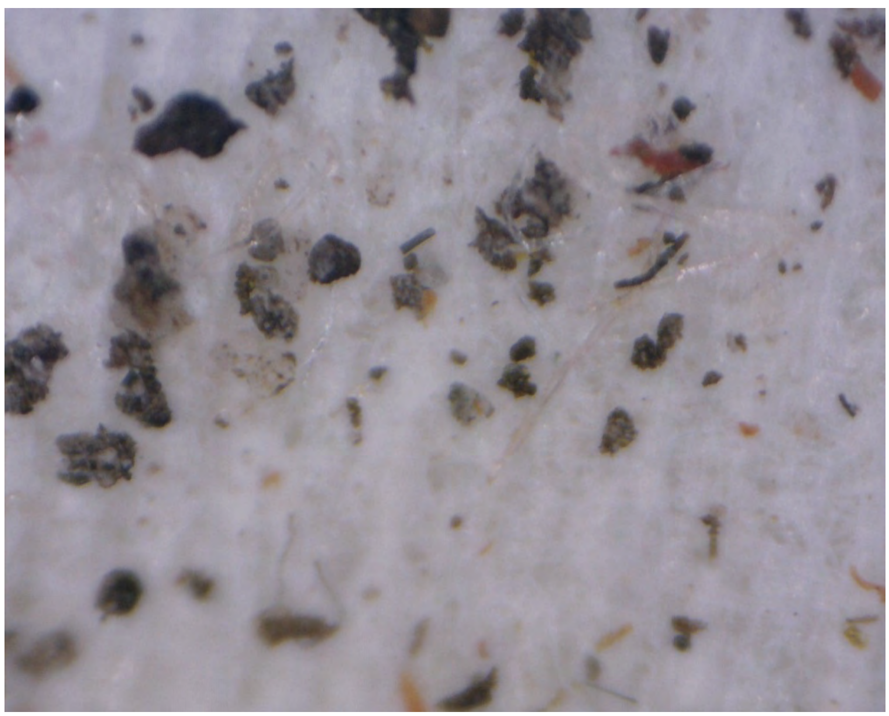

Figure 6. High Magnification (150X) Image of Particles Shaken from Andersen AFB Fire Hose Samples

4.1.2. Live Fire Sampling

Square coupons cut from the woven outer-layer fabric normally used for "structural bunker" suits were exposed to smoke in the Fire Hanger facility during graphite-epoxy composite fires. Some coupons were observed with the Celestron™ 44302 microscope. The structural bunker material exhibited small holes between the thread strands comprising the material, and the edges of cut coupons were subject to loss of threads, which could influence the final weight of a coupon observed with a balance, and which could deposit additional fibers (threads) on the coupons.

In one experiment, the coupons were weighed before and after the fire in the expectation that they would gain weight from smoke and other particulates that were deposited in the fire. This experiment essentially failed when nearly all of the coupons lost weight upon exposure to the fire and smoke, and some gained weight upon being cleaned with a vacuum cleaner. For this experiment, an ordinary vacuum cleaner was used. The unexpected weight behavior may have been due to moisture absorption or release in the samples, superimposed on weight changes due to smoke and fiber contamination. The weighed coupon experiment was attempted in association with a fire which was extinguished shortly after ignition. Comparatively few particles were visible on the coupons, and it is possible that the fire only produced a low concentration of airborne particulates in the test facility.

In a subsequent experiment, the fire was permitted to burn out the liquid jet fuel, and the coupons were not disturbed until no flames were visible anymore on the composite material. These coupons appeared to collect more smoke particles. The masses of the coupons were not measured for this experiment. Photomicrographs of typical structural bunker coupons are shown in Figure 7. The upper left image shows the low-magnification range image, and the upper right image is the high-magnification (150X) image of the same coupon. The coupons were cleaned with an ordinary vacuum cleaner and re-examined. The lower left image is the low-magnification range, post-cleaning image of the same coupon and the lower right image is the high-magnification, post-cleaning image. Other images, not shown, indicated that strands of the fabric polymer material extended above the "surface" of the fabric and trapped some additional smoke particles, but obtaining these images required shifting the microscope focus. The weave holes in these images prevented effective recognition of the smoke particles for automated measurement and counting purposes. As such this experiment proved suitable only for gross information on the contamination of the samples.

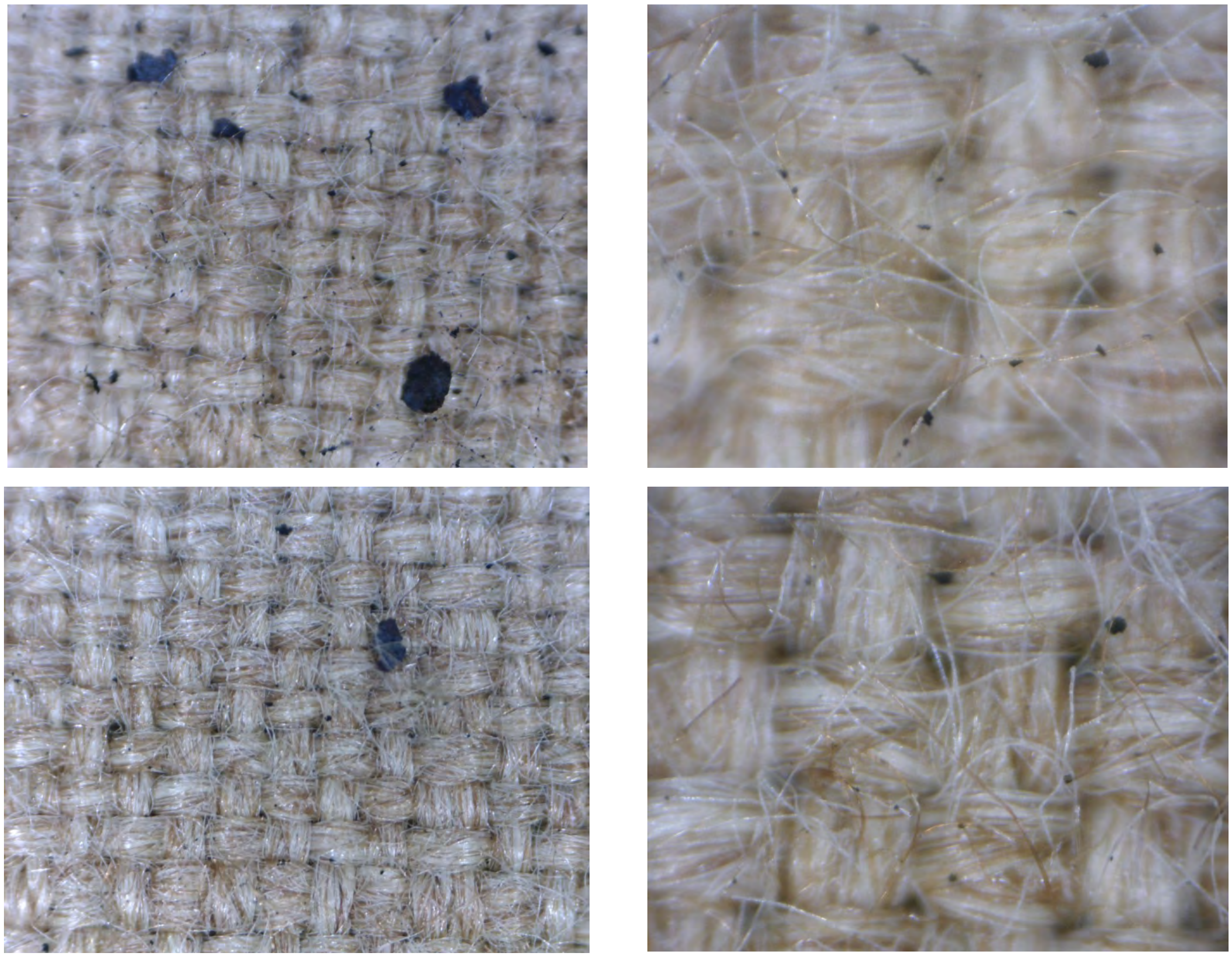

Figure 7. Photomicrographs of a Structural Bunker Coupon, After Exposure to a Composite Fire (Top Images) and After Cleaning (Bottom Images)

Fabric intended for use as the outer layer of proximity bunker suits, also called “silver suits” or proximity bunkers was cut into 2-inch square coupons and exposed to composite material test fires along with the structural bunker coupons as described above. Figure 8 shows a typical proximity bunker coupon exposed in the full-duration composite fire. The upper left photomicrograph images a field of view from the coupon in the low magnification range, after the fire but before cleaning. The upper right image in Figure 8 shows the high magnification (150X) image of a section of the coupon. The coupons were cleaned with an ordinary vacuum cleaner and imaged again. The lower left image is the low magnification photomicrograph of the coupon after cleaning, and the lower right image is the high magnification (150X) view. Human inspection of these photomicrographs reveals smoke particles on the post-burn images, and not on the post-cleaning images. However, the image analysis failed in this case due to the light-and-shadow effects produced by the silver-colored surface and undulating texture underneath the surface. The Celestron™ 44302 offered no means of adjusting the lighting except to adjust the microscope position relative to the coupon, as the light was provided by a circular array of 6 white LEDs arranged in a circle around the objective lens, and the LEDs were lit any time the microscope was plugged into its host computer.

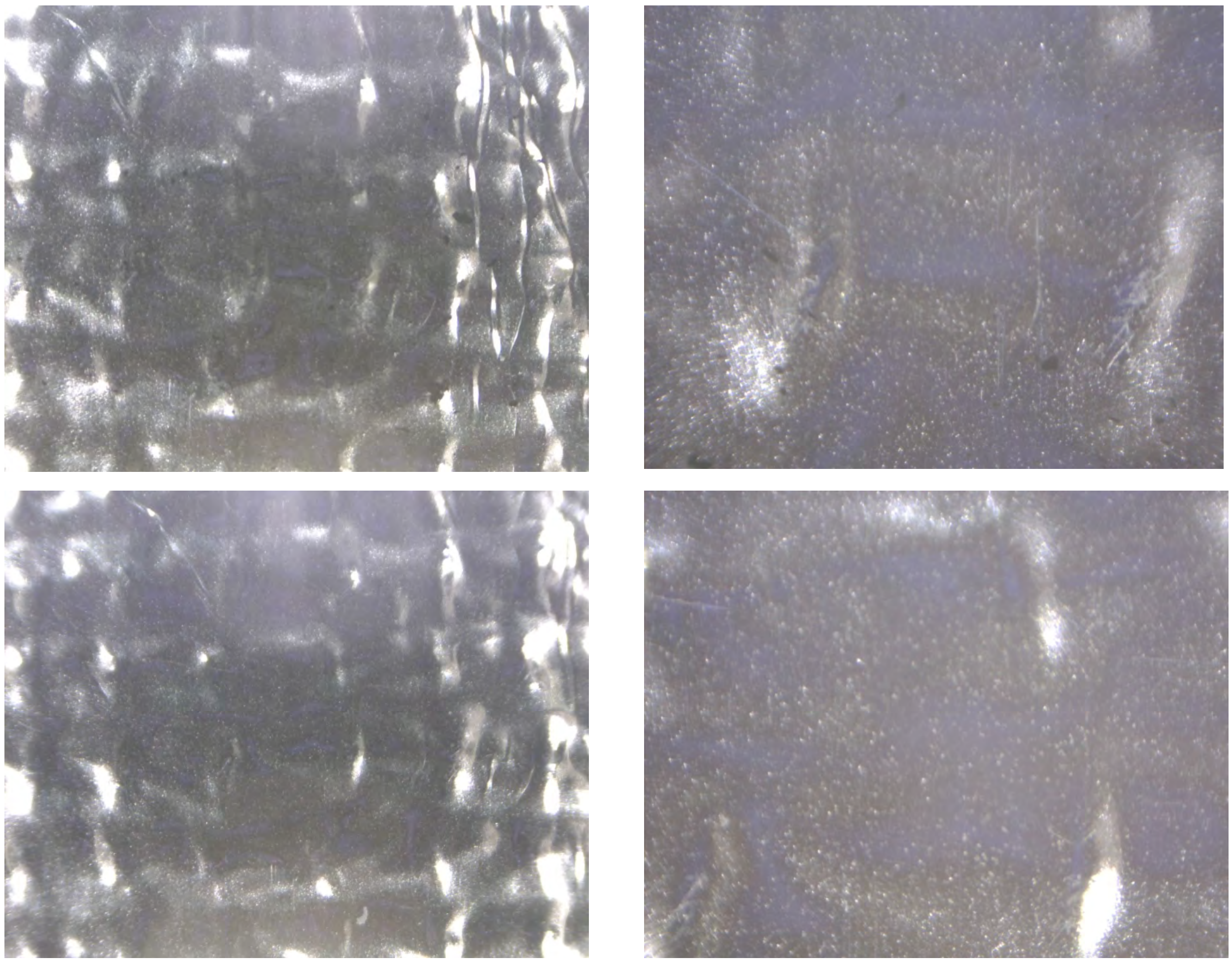

Figure 8. Photomicrographs of a Proximity Bunker Coupon. The Upper Images are Post-Fire and the Lower Images are Post Cleaning

4.2. Sticky-Pad Sampling and Microscopy

Due to the difficulties noted above, a small number of bunker material coupons were soiled using a predecessor of the bake-and-shake procedure, using a plastic pail and cover as the exposure facility in place of the cardboard box eventually used. These coupons included the proximity bunker outer (silvered) layer, the structural bunker suit outer layer, and two inner layers common to most bunker suits. These exposed coupons were sent to external experts for microscopic analysis. From this, the sticky pad sampling was suggested.

White opaque pads and clear-on-white pads were available for sampling. A photomicrograph from a prepared slide using the clear pads is shown in Figure 9. The left frame was taken with lower magnification, using a 4X objective lens for an estimated 40X magnification. The right frame was taken using a 10X objective lens for an estimated 100X magnification. This slide was prepared from a silver-suit coupon, contaminated with the bake-and-shake technique and sampled using a clear sticky sheet which was then covered with a slide cover slip and placed on a glass microscope slide. The slide was viewed and photographed with transmitted light on the SPI 1864 microscope and Motic™ microscope camera. Although the combustion particulates are clearly visible, bubbles are evident around some of the particles and forming at random in the field of view. These bubbles could not be eliminated from detection with the image analysis software. The red rectangle visible in both frames was accidently inserted through the Motic™ software and was removed for subsequent quantitative experiments.

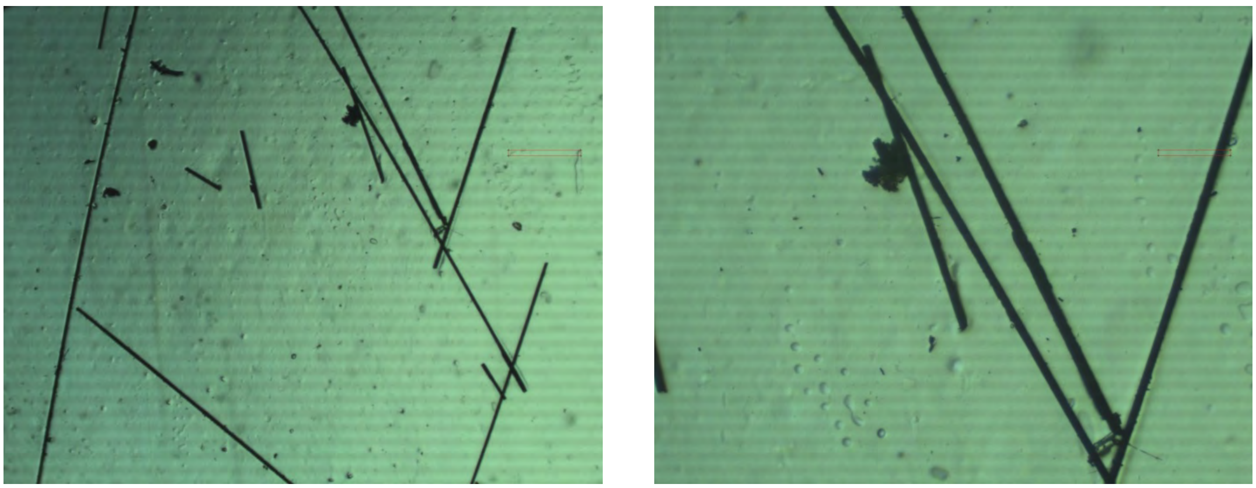

Figure 9. Photomicrographs from a Close-Approach Bunker Coupon Sampled with a Clear Sticky Sheet and Imaged with Transmitted Light

The bubbles were less visible using a white sticky pad and overhead lighting. To do this, the sticky pad and trapped particulates were affixed to the bottom-side of a microscope slide and the particulates were viewed through the glass of the slide. The slide was cleaned with a kimwipe™ prior to sampling, and the top of the slide could be cleaned off as necessary to prevent ordinary dust from interfering with the particle counts.

The combination SPI 1864 microscope, Moticam 2500, and MATLAB Image Processing Toolbox was capable of detecting objects down to the size of a single pixel on the digital images. At the lower (40X) magnification, the pixel size was ~1.3 µm and at the higher (100X) magnification the pixel size was ~0.5 µm. Careful examination showed the digital images were slightly cropped from the human-eye images, so the actual image magnifications exceeded the nominal magnifications. However, comparisons of magnification between a microscope eyepiece image and an image projected on a computer display are somewhat arbitrary in nature. The original Moticam images were of higher resolution than the .jpg images and might have reduced the pixel size accordingly if software were available to utilize them.

There was considerable variation among the particulates seen on any given coupon and any given type or class of coupon. Figure 10 shows selected photomicrographs. The upper left frame, shows the field of view with the largest measured particulate area from the exposed, non-cleaned coupons while the upper right frame, shows the lowest particulate area for the exposed, non-cleaned coupons. The lower left frame, shows the field of view from the unexposed coupons with the greatest measured particulate area while the lower right frame, shows the field of view from the unexposed coupons with the lowest measured particulate area. Particulates and threads were seen on a few of the unexposed coupons and some obviously originated from unraveled bunker fabric. The lower right field of view is a clean frame, without detected particulates.

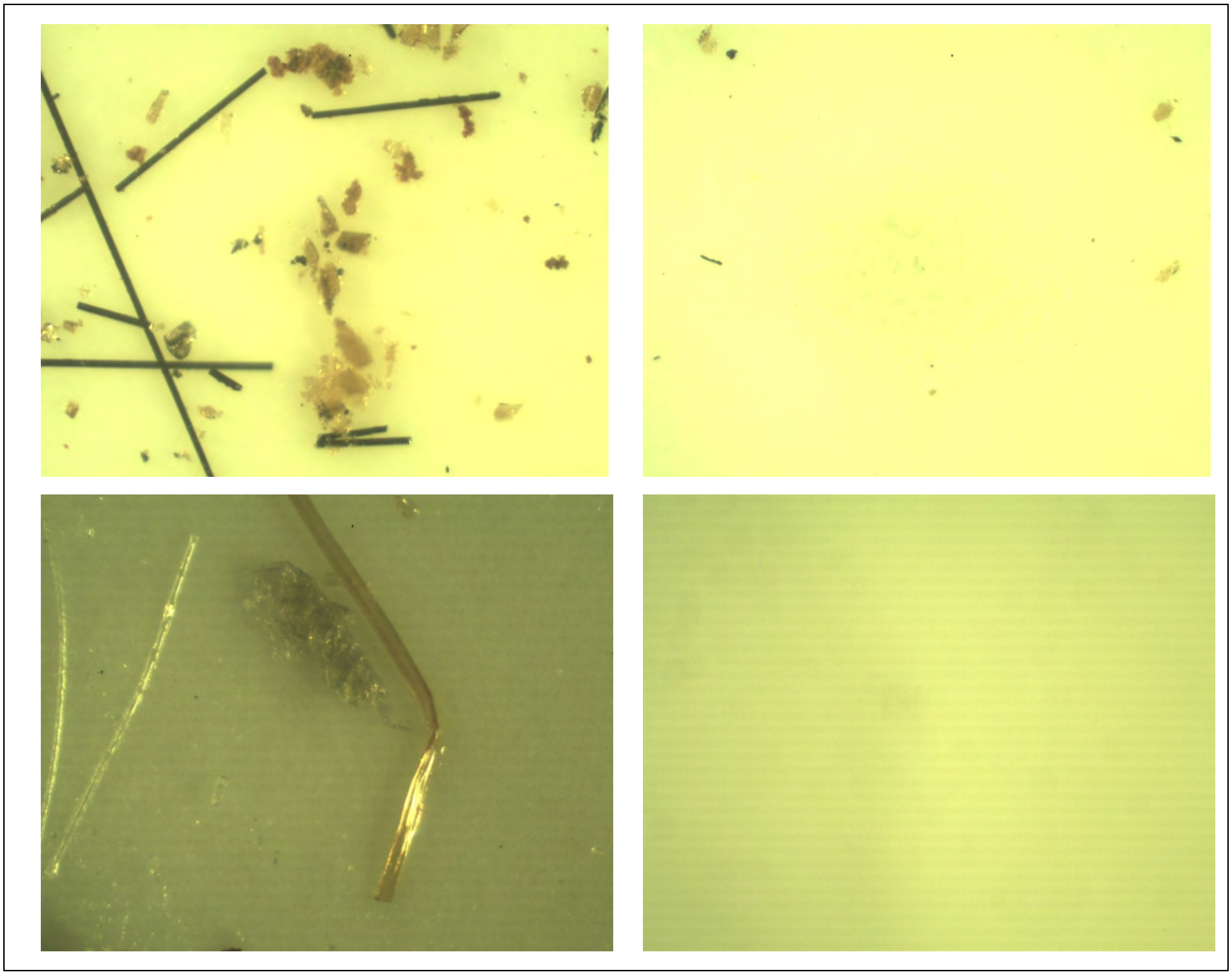

Figure 10. Selected Photomicrographs from Particulates Sampled from Proximity Bunker Material, Contaminated from Burned Composite, and Sampled with White, Opaque Sticky Pads

4.3. Cleaning Experiment

A supply of ~1-inch × 1-inch coupons were cut from proximity bunker outer-layer fabric. These were pinned to a cardboard sheet in an array of 9 rows of 6 coupons each. Six unexposed silver-suit coupons were removed from all rows of the sample array and sampled with clean sticky-pad layers to measure the level of contamination to the coupons from general handling in the laboratory. The remaining coupons were exposed to 9.83818g of the burned composite material by gently sloping the exposure chamber through all four directions at a gentle angle (~30 deg.) and gently agitating for about 8 min. After the agitation period all composite material was removed from the exposure chamber. Seven contaminated, but non-cleaned, coupons were sampled and evaluated to provide a pre-cleaning baseline for the coupons, and an additional four non-cleaned coupons were evaluated after all cleaning experiments were accomplished, to determine if some of the contaminants might escape during the time required to accomplish the experiment. Cleaning experiments were conducted on single rows of coupons, cut apart from the remaining sheet of exposed coupons, except for the vacuum cleaning trial which used coupons from two rows. Two coupons from the vacuum cleaning row were sucked into the vacuum cleaner nozzle despite the attachment pins, so three coupons were selected from the adjacent, non-cleaned row, and cleaned with the vacuum cleaner, although one of these coupons was also detached and ingested by the vacuum cleaner. Vacuum cleaning was observed with six valid coupons. Seven coupons were cleaned with water washing and examined. Seven coupons were cleaned with Lysol decontaminating wipes and observed. Seven coupons were cleaned with the Evercare lint roller.

The evaluated number and area of contaminants, classed by cleaning methods, are summarized below in Table 1. The averages were calculated from the particle area over all fields of view for a cleaning class. The median tabulated was the median number of particles from all fields of view for a cleaning class. The number *n* given for each class is the total number of all fields of view for the cleaning class. The number of degrees of freedom, *df*, used for Students-t and other comparison testing, was calculated as the number of observations, *n*, minus a degree of freedom for each coupon. The Cleanability was calculated as in Equation 2.

Table 1. Summary of Particle Counting and Cleaning Experiments

Sample Type	Median Number of Particles	Average Particle Area (pixels)	Pooled *s* (pixels)	Cleanability	Total *n*	Total *df* (v)
Blank Sticky Pads	0	1.77E+01	5.61E+01		42	37
Unexposed Coupons	1	6.84E+03	2.11E+04	100.00	52	46
Exposed, Non-cleaned Coupons (Set 1)	45	5.44E+04	3.67E+04	0.00	63	56
Brush Cleaned Coupons	21	2.40E+04	1.97E+04	63.81	65	58
Water Washed Coupons	11	5.40E+03	1.40E+04	103.02	62	55
Vacuum Cleaned Coupons	37	9.41E+03	1.58E+04	94.59	54	48
Lysol Wiped Coupons	7	1.59E+04	1.86E+04	80.95	55	49
Lint Rolled Coupons	9	3.85E+03	6.75E+03	106.27	65	58
Exposed, Non-cleaned Coupons (Set 2)	51	3.76E+04	2.29E+04	35.31	36	32

Student's-t testing was applied to test the differences between the mean results from the tested cleaning methods. The null hypothesis between two means can be rejected if the t-statistic between the two means is greater than the tabulated t-value for a given level of significance. The t-statistic calculations and comparison results are summarized in Table 2. The results in Table 2 indicate that the two sets of exposed, non-cleaned coupons do not differ significantly, but water-washed coupons and lint-rolled coupons differed significantly from the exposed, non-cleaned coupons at the 95% confidence level. The second set of exposed, non-cleaned coupons was not tested against the cleaned coupons. The second set of tests (columns 11-16) indicates that at the 90% confidence level (significance 0.10), both sets of non-cleaned coupons differ significantly from the unexposed coupons and none of the means from the cleaning techniques differ significantly from the unexposed coupons.

Table 2. Statistical Results from Coupon Data

T-Calculations, Testing of Significance.															
						Comparison Cleaned vs. Non-cleaned - Exposed				Comparison of Exposed or Cleaned vs. Unexposed					
Sample Type	Median Number of Particles	Average Particle Area (pixels)	*s*-Pooled for the Cleaning Class (pixels)	*n*	*df*	*s*-Pooled for means comparison (pixels)	Calculated *t*	*t* from table	t>t (0.05)? (i.e. Significant?)	*s*-Pooled for means comparison (pixels)	Calculated *t*	*t* from table	t>t (0.05)? (i.e. Significant?)	T(0.10) 90% confidence from table	t>t(0.10) (90% confidence)
Blank Sticky Pads	0	0.00E+00	5.61E+01	42	37										
Unexposed Coupons	1	6.84E+03	2.11E+04	52	46										
Exposed, Non-cleaned Coupons (Set 1)	45	5.44E+04	3.67E+04	63	56					30659.47	-1.551	1.645	no	1.282	yes
Brush Cleaned Coupons	21	2.40E+04	1.97E+04	65	58	29299.72	1.035	1.645	no	20324.73	-0.847	1.645	no	1.282	no
Water Washed Coupons	11	5.40E+03	1.40E+04	62	55	27851.63	1.759	1.645	yes	17575.86	0.082	1.645	no	1.282	no
Vacuum Cleaned Coupons	37	9.41E+03	1.58E+04	54	48	28980.73	1.552	1.645	no	18578.09	-0.138	1.645	no	1.282	no
Lysol Wiped Coupons	7	1.59E+04	1.86E+04	55	49	29654.97	1.298	1.645	no	19850.67	-0.456	1.645	no	1.282	no
Lint Rolled Coupons	9	3.85E+03	6.75E+03	65	58	26163.01	1.931	1.645	yes	14912.95	0.200	1.645	no	1.282	no
Exposed, Non-cleaned Coupons (Set 2)	51	3.76E+04	2.29E+04	36	32	32353.15	0.519	1.645	no	21840.84	-1.408	1.645	no	1.282	yes

5. CONCLUSIONS

Water washing and the sticky lint roller were the best techniques of those tested for removing particulate contaminants from proximity bunker fabrics. Vacuum cleaning provided a reasonably high cleanability, but the cleaned coupons did not differ from the contaminated coupons at the 95% confidence level, thus the effectiveness of vacuum cleaning was not demonstrated as clearly as water washing and lint rolling.

These tests indicate the coupons, representing proximity bunker fabric, are substantially cleaned by the tested techniques, particularly water-washing and by use of a lint roller. Both techniques should be readily available to firefighting flights during post-burn decontamination and when supporting aircraft recovery teams.

The quantitative cleaning data addressed particulates ranging from ~0.5 μm to arbitrarily large fibers and chunks. The quantitative data contained in this report were from specimens contaminated by post-fire contact with burned composite materials. Experimental fires were only available prior to the refinement of the techniques for quantitative sticky-sheet sampling and image analysis for particulates. Additional quantitative experiments should be conducted to test cleaning of clothing and equipment exposed to live-fire smoke and particulates.

This experiment concentrated on particulate contamination. Further experiments will be needed to examine organic chemical contamination on the surface of the equipment, and these will require other, non-microscopic techniques.

None of the tests performed in this experiment prove a garment or equipment cleaned by these techniques can be guaranteed safe, i.e. harmless, for future use.

6. RECOMMENDATIONS

Water washing and lint roller cleaning appear to be the most effective for removing composite smoke particles and fugitive fibers from soiled bunker clothing. Vacuum cleaning appears effective enough to recommend where water washing or lint rollers are not usable, as may be the case where equipment is not waterproof, or in the case where lint rollers are unavailable or unaffordable. Brushing and cleaning with decontaminating wipes do not appear as effective as the other techniques and are not recommended.

The experiments performed cannot recommend combinations of cleaning techniques as the experiments to date studied only single cleaning techniques and no combinations. A number of research questions remain as to the degree of contamination expected on clothing and equipment exposed to composite fires. Additional observations are needed to extend the experiments reported here to actual fire particulates. Additional research is needed to measure polynuclear aromatic hydrocarbons and other potential organic chemical carcinogens and toxins on fire-exposed coupons. Quantitative organic analysis techniques could allow a clearer confirmation of coupon contamination and cleaning, although these will require different instrumentation to measure than the camera used in the current study. Soap-and-water has been suggested in place of simple water for cleaning some clothing items,[20] but this suggestion was not received in time to act in this series of experiments. Soap-and-water cleaning is also seldom effectively done entirely by hand, and not all base fire departments appear to have access to industrial laundry facilities.

LIST OF SYMBOLS, ABBREVIATIONS, AND ACRONYMS

ACM	advanced composite material
A_c	area of contaminant particulates per field of view, on a cleaned coupon
AFCESA	Air Force Civil Engineering Support Agency
AFRL/RXQ	Air Force Research Laboratory, Airbase Technologies Division
ALI	acute lung injury
A_o	area of contaminant particulates per field of view, on an original coupon
A_s	area of contaminant particulates per field of view, on a soiled (or exposed) coupon
$B_{x,y}$	blue value of the RGB color pixel at image coordinate x,y
C	cleanability value
df	statistical degrees of freedom
FAA	Federal Aviation Administration
GC/MS	gas chromatography/mass spectrometry or a gas chromatograph/mass spectrometer instrument
$GS_{x,y}$	grayscale value of the RGB color pixel at image coordinate x,y
$G_{x,y}$	green value of the RGB color pixel at image coordinate x,y
n	sample number or count
NASA	National Aeronautics and Space Administration
PAN	polyacrylonitrile
PPE	personal protective equipment
RAF	Royal Air Force (UK)
$R_{x,y}$	red value of the RGB color pixel at image coordinate x,y
s	standard deviation estimate from a sample
SPME	solid phase microextraction
t	student's *t*-statistic
ν	statistical degrees of freedom, mathematical symbol for df
$\bar{x}$	arithmetic mean or average of quantity x

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